Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP)

Powder Coatings - Generic Testing and Quality Assurance Protocol

Draft

February 17, 1998

Prepared by
National Defense Center for Environmental Excellence (NDCEE)

Operated by Concurrent Technologies Corporation

Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP)

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Submitted by

Concurrent Technologies Corporation
1450 Scalp Avenue
Johnstown, PA 15904

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1.0 INTRODUCTION

1.1 Purpose of the Generic Testing and Quality Assurance Protocol

The primary purpose of this document is to establish the general procedure for powder coatings testing. The secondary purpose is to establish the general format and guidelines for powder coatings Testing and Quality Assurance Project Plans (TQAPPs).

Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP) project level TQAPPs establish specific data quality requirements for all technical parties involved in the project. A defined format, as described below, is to be used for all ETV CCEP TQAPPs to facilitate independent reviews of project plans and results, and to provide a standard platform of understanding for stakeholders and participants.

1.2 Quality Assurance Category for ETV CCEP

Projects conducted under the auspices of the ETV CCEP will meet or exceed the requirements of the Category II Quality Assurance Project Plan (600/8-91/004, February 1991) preparation aid established by the Environmental Protection Agency's (EPA) National Risk Management Research Laboratory (NRMRL). This protocol is intended to ensure that the project results are compatible with and complementary to similar projects. ETV CCEP coatings technology TQAPPs adapted from the guidelines discussed in the EPA preparation aid and this plan, would contain sufficient detail to ensure that measurements are appropriate for achieving project objectives, that data quality is known and that the data are legally defensible and reproducible.

1.3 Logic and Organization of the Protocol Document

This coatings technology protocol document contains the sections outlined in the EPA Category II QAPP guidance document. As such, this protocol identifies processes to be used, test and quality objectives, measurements to be made, data quality requirements and indicators, and procedures for the recording, reviewing and reporting of data.

The major technical sections to be discussed in this protocol are as follows:

- Project Description
- Project Organization and Responsibilities
- Quality Assurance (QA) Objectives

- Site Selection and Sampling Procedures
- Analytical Procedures and Calibration
- Data Reduction, Validation and Reporting
- Internal Quality Control Checks
- Performance and System Audits
- Calculation of Data Quality Indicators
- Corrective Action
- Quality Control Reports to Management
- References
- Appendices

1.4 Formatting

In addition to the technical content, this protocol also contains standard formatting elements required by EPA Category II guidelines and *CTC* deliverables. Standard format elements include, at a minimum, the following:

- Title Page
- QA Project Plan Approval Page
- Distribution List
- Table of Contents (with an explanation of any deviations from Category II required elements)
- Document Control Identification (in the plan header)

Section No. _______
Revision No. _______
Date: _______
Page: ___ of ___

1.5 Approval Form

Key *CTC* personnel involved with the ETV CCEP will indicate their agreement and common understanding of the project objectives and requirements by signing the TQAPP Approval Form for each powder coating tested. Acknowledgment by each key person indicates commitment toward implementation of the plan. Figure 1 shows the Approval Form format to be used.

APPROVAL FORM

Date Submitted: ________________ QTRAK No.: ______________

Revision No.: __________________ Project Category: ______________

Title: ____

Project/Task Officer: ________________________________

CTC/Address/Phone No. ______________________________

Interagency
Agreement No.: _______________ Task No.: _________ Duration: _________

APPROVALS

CTC Project/Task Manager	Signature	Date
CTC QA Officer	Signature	Date
NRMRL/APPCD Project/Task Officer	Signature	Date
NRMRL/APPCD QA Officer	Signature	Date

CTC - Concurrent Technologies Corporation
NRMRL - National Risk Management Research Laboratory
APPCD - Air Pollution Prevention and Control Division

Figure 1. Testing and Quality Assurance Project Plan Approval Form

2.0 PROJECT DESCRIPTION

2.1 General Overview

Organic coatings are used by many industries for protection and decoration of their products. Coatings with organic solvents contribute nearly 20 percent of total stationary area source volatile organic compound (VOC) emissions, as well as a significant percentage of air toxic emissions. Powder coatings are continually being developed by many sources in an effort to reduce any detrimental effects to the environment. Often these powder coatings are slow to penetrate the market because potential users, especially an ever-growing number of small companies, do not have the resources to test powders on their particular application and may be constructively skeptical of the powder coating provider's claims. If an unbiased, third party facility could provide pertinent test data, environmentally friendlier coatings would penetrate the industry faster and accelerate environmental improvements.

The ETV CCEP, a joint venture of the US Environmental Protection Agency (EPA) and Concurrent Technologies Corporation (*CTC*), in conjunction with the National Defense Center for Environmental Excellence (NDCEE) in Johnstown, Pennsylvania, has been established to provide just such unbiased, third party data. The ETV CCEP has been tasked to develop and subsequently use a standardized protocol for verifying performance characteristics of powder coatings.

To maximize its exposure to the coatings industry, the data from the verification testing will be made available over the internet on the EPA's Environmental Technology Verification Program website (http://www.epa.gov/etv/) under the P2/Innovative Coatings and Coating Equipment Pilot, as well as through other sources (e.g., publications, meetings, etc.). This will help establish the ETV CCEP's reputation in the private sector. A long range goal of this initiative is to grow the Program's reputation so that it becomes a vital resource to the industry and thus self-sustaining through private support. This is in addition to its primary objective of improving the environment by rapidly introducing more environmentally friendly coating technologies into the industry.

2.1.1 Demonstration Factory Testing Site

CTC has been tasked under the NDCEE Program to establish a demonstration factory capable of prototyping processes that will reduce or eliminate hazardous wastes used in manufacturing. In order to speed the transition of environmentally-friendly processes to the manufacturing base, *CTC* offers the ability to test processes and products on full-scale, commercial equipment. This demonstration factory is a major national asset. It includes a combination of organic finishing, cleaning, stripping, inorganic finishing, and recycle/recovery equipment. The organic coating equipment in the demonstration factory will be available for the pilot-scale testing used in this project. Specifically, these include surface pretreatment, powder coating, electrocoating, liquid spray booths, and conventional and infrared cure ovens. Ancillary equipment from plating, non-halogenated cleaning and non-chromate conversion coating may also be required. A layout of the *CTC* Demonstration Factory is shown in Figure 2 below. A layout of the organic finishing line is shown schematically in Figure 3.

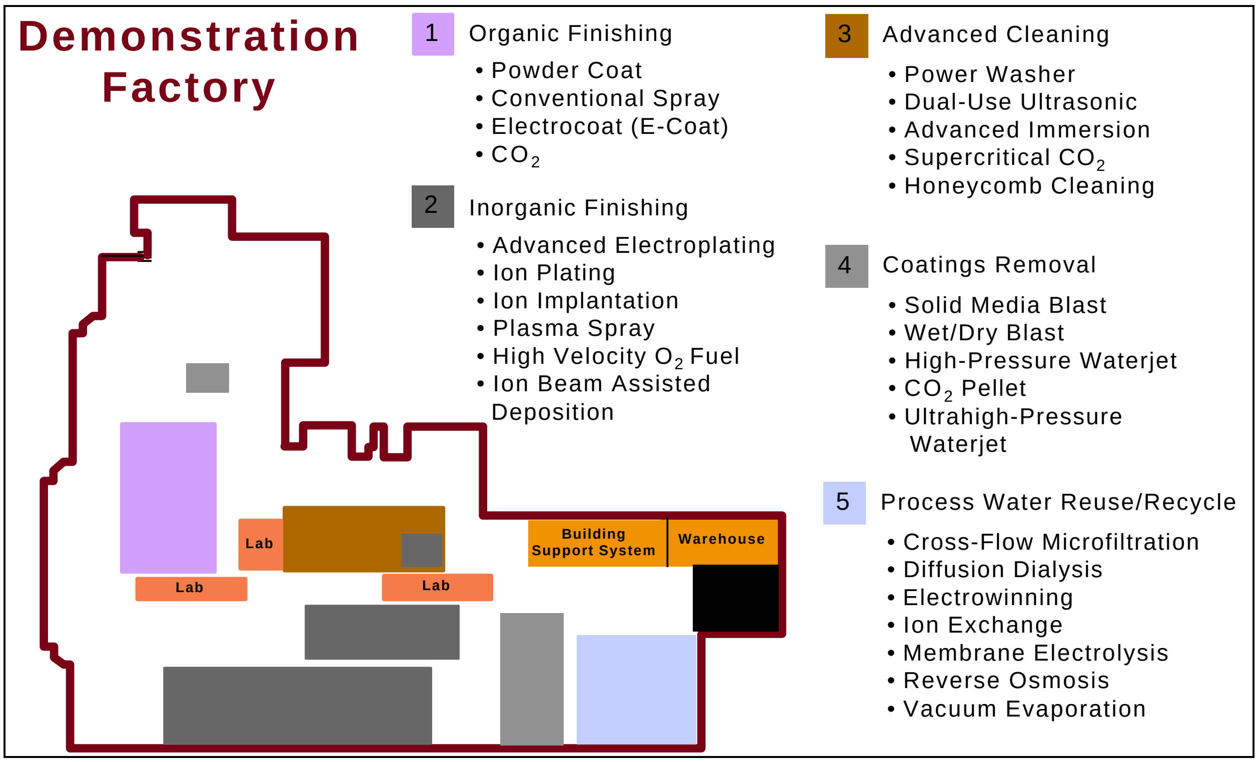

Figure 2. *CTC* Demonstration Factory Layout

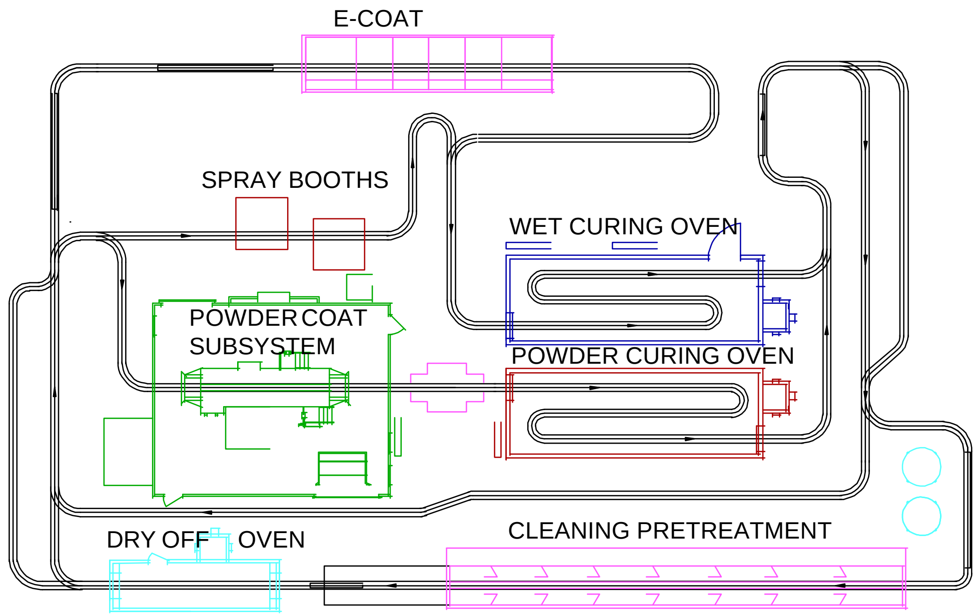

Figure 3. ***CTC*** **Demonstration Factory Organic Finishing Line**

It may be necessary to test a particular technology away from *CTC*. If such is the case, this testing will be done under the control and close observation of ETV CCEP personnel, and always in compliance with the TQAPP. This includes any and all Quality Assurance (QA) and appropriate laboratory analyses.

2.1.2 Laboratory Facilities

In support of the demonstration factory coating processes, *CTC* maintains extensive state-of-the-art laboratory testing facilities. These laboratory facilities are used for the measurement and characterization of processes and specimens as well as for bench-scale coating technology evaluations. Table 1 lists the various testing and evaluation laboratories, as well as representative equipment holdings, relevant to ETV CCEP projects.

Table 1. Testing Laboratories and Representative Laboratory Equipment Holdings

Laboratory	Focus	Laboratory Equipment
Environmental Testing	1) Identification and quantification of biological, organic, and inorganic chemicals and pollutants to all media. 2) Industrial process control chemical analysis.	Hewlett Packard 5972A GC/MS Varian Liberty 110 Sequential ICP P-E 4100ZL Graphite Furnace Mitsubishi GT06 Autotitrator P-E Headspace GC/ECD/FID TOC/Flashpoint/pH/Conductivity Graseby 2010 Isokinetic Stack Analyzer Graseby 2800 VOST Stack Sampler Questron Q-Wave 1000 Microwave Leeman PS200/AP200 Mercury Stations Millipore TCLP/ZHE Extraction Station Lachat Quickchem Flow Injection Analyzer
Destructive and Non-Destructive Evaluation	Evaluation of product and process performance, and surface cleanliness.	Optically Stimulated Electron Emission X-ray/Magnetic/Eddy Current Thickness Salt Spray Corrosion Chamber Microhardness/Tensile/Fatigue/Wear
Materials and Mechanical Testing	Measurement of service and processing material and mechanical properties.	Noran and CAMScan Electron Microscopes Leco 2001 Image Analysis System Nikon and Polaroid Light Optical Microscopes EDAX Energy Dispersive Spectrometer Single Crystal Imaging Metallography Polishing/Grinding/Etching MTS Machines Tinius Olsen Testers Impact Testers
Powder Metallurgy	Investigation of Powder Properties.	Horiba LA900 Laser Particle Size Analyzer Autopore II 9020 Mercury Porosimeter Accupyc 1330 Pycnometer Gemini II 2370 Surface Area Analyzer
Intelligent Processing of Materials	Development and evaluation of embedded process sensors.	TEC Model 1600 Stress Analyzer Spectraphysics Argon & ND:YAg Lasers Resonance Frequency System
Risk & Environment Analysis	Management, monitoring and evaluation of material and process alternatives from health and safety perspective.	Biosym: molecular modeling software MOPAC, Extend, HSC Chemistry, Riskpro, Sessoil, GIS
Calibration Laboratory	Calibration of equipment, sensors, and components to nationally traceable standards	Transmation Signal Calibrator (milliamps, millivolts) Thermacal Dry Block Calibrator (Temperature) Druck Pressure Calibrator (Pressure) Fluke Digital Multimeter (Voltage)

2.1.3 Statement of Project Objectives

The overall objective of the ETV CCEP is to verify performance and pollution prevention characteristics of coatings and coating equipment, and to make the results of the testing available to prospective coatings users. The objective of this particular protocol is to verify the performance of specific powder coatings. Whenever one exists, accepted American Society for Testing and Materials (ASTM) methods will be used for analyses.

2.2 Technical/Experimental Approach and Guidelines

The following tasks are planned for this project (see estimated schedule in Section 2.3, Table 6):

1. Conduct initial stakeholders meeting
2. Investigate/identify/prioritize focus areas
3. Draft and revise Commerce Business Daily (CBD)/Request for Technology (RFT) for powder coatings
4. Approval and issuance of CBD/RFT
5. Draft and revise Generic Testing and Quality Assurance Protocol for powder coatings
6. Receive/review responses to CBD/RFT
7. Approval and issuance of final Generic Testing and Quality Assurance Protocol
8. Stakeholder conference call to choose pertinent CBD/RFT responses for verification testing
9. Produce and obtain approval for specific TQAPPs for each powder coating to be tested
10. Verification Testing
11. Prepare test report
12. Approval of test report by EPA
13. Verification Statement - Issued by EPA

Each TQAPP is greatly dependent upon the particular powder coating to be tested. Regardless of the specific powder coating tested, there are certain overall guidelines and procedures which will be applied to the TQAPP.

Table 2 lists these overall guidelines and procedures. Table 5 gives the set of tests typically performed at *CTC* to determine the product quality of a powder coating. It should be noted that these tables do not intend to be all-inclusive, and TQAPPs for specific powder coatings may not include tests and procedures listed.

Table 2. Overall Guidelines and Procedures to be Applied to the Generic Protocol

- A detailed description of each part of the test will be given. This will include a detailed Design of Experiments, and a schematic diagram of testing to be performed (see Figure 4).

- Critical and Non-critical factors will be listed. Non-critical factors will be held constant throughout the testing. Critical factors will be listed as Control (process) factors or Response (powder coating product quality) factors (see Section 2.28 below).

- The Test Protocol will identify the testing site.

- Regardless of where the testing is done, all testing will be under the control and close supervision of *CTC* representatives to ensure the integrity as third party testing.

- Regardless of where the testing is done, the QA portion of the Test Protocol will be strictly adhered to.

- A statistically significant number of samples will be analyzed for each critical response factor (see Table 5) up to a maximum of 10 samples each (2 samples from each of 5 runs). This limit is due to budgetary concerns and may be extended at an added cost to the technology provider. Variances (or standard deviations) of each critical response factor will be reported.

2.2.1 Test Approach

The following approach will be used in the test protocol.

- Performance parameters to be verified will be determined
- A standard test panel and product will be coated which will enable thorough testing of the powder coating's performance
- Powder manufacturers will provide the coatings and optimum settings for application, and curing, and
- A statistically valid test program that efficiently accomplishes the required objectives will be utilized.

2.2.2 Standard Test Product and Panels

The standard test product to be used during testing is shown in Appendix A. This standard test product can be described as three adjacent perpendicular planes of a cube. It is made of steel and is 12 inches long on each edge and 1/16 of an inch thick (approximately 2.6 pounds in weight). It has two 1/4 inch holes punched in two adjacent sides for hanging. This standard test product was chosen because it will be very effective in evaluating the Faraday Cage and wrap around effects of the powder coatings tested. Film thickness measurements will be taken from the locations on the test specimen noted in Appendix A to test for film thickness uniformity. This standard test product will be used in the evaluation of powder coating performance when spraying the inside and/or outside surfaces of a three-dimensional object. The standard test product will be racked in a manner that allows the paint spray pattern equal access to all surfaces facing the coating equipment (see Appendix C, Apparatus Set-Up).

In addition to coating the standard test product, test panels will be coated and used to determine coating quality. The test panels to be used are flat, steel panels, 12" x 4". A hole in one end of the panels will be used to hang each panel from a conveyor rack during testing. Other parts can be coated to satisfy the technology provider's request at an additional cost to the technology provider. The organic finishing line in the Demonstration Factory at *CTC* can accommodate parts up to 4' x 4' x 3' weighing up to 250 pounds.

As a preparation for coating, the parts will receive a zinc phosphate pretreatment. The pretreatment portion of the organic coating line in the *CTC* Demonstration Factory is a staged operation. During pretreatment, the standard part or panel will receive an alkaline clean followed by a DI water rinse. Then the zinc phosphate is applied followed by another DI water rinse and then a dry off stage. If a sealer is called for in the individual TQAPP, it would be applied before the dry off stage, and would be followed with a DI water rinse before going to dry off. As the Demonstration Factory is a continuous coating line, the time between dry off and the coating booth will depend only on the production rate allowed by the coating equipment and the powder coating itself. Similar powder coating technologies will receive the same pretreatment. Because a consistent pretreatment weight per unit area is historically an important factor for powder coating performance, one panel from each rack of panels will be taken and tested to assure consistency as part of the design of experiments.

2.2.3 Powder Coating Apparatus

The organic finishing line at *CTC*'s Demonstration Factory will be used to apply the powder coatings to the standard test products and panels. All products and panels will be pretreated with zinc phosphate, unless specified to have no pretreatment, prior to entering the powder coating subsystem. A thickness range will be designated for each powder coating, as well as cure temperature and time. Four automatic corona type guns will apply the powder to the test products and panels.

2.2.4 Reliability Check of the Powder Coating Equipment

A standard powder coating will be chosen for each powder coating focus area. A history of the coating quality of the standard powder coating will be developed (if one does not already exist). A standard powder coating run will be made immediately after each set of powder coating test runs and the results compared to the historical data to assure the quality of the operation of the spray equipment.

2.2.5 Determination of VOC and HAP Content of the Powder Coating

A determination of the VOC and HAP content of the powder coating will be done under curing conditions (time and temperature). At the time of the writing of this generic TQAPP there is no satisfactory test method for doing this to meet the objective of the ETV program and industry stakeholders. The US EPA is currently developing a satisfactory test method and it will be applied to this TQAPP as soon as it is developed.

2.2.6 Design of Experiment

This protocol will determine the performance of powder coatings submitted in response to the associated CBD or RFT. A mean value and variance (or standard deviation) will be reported for each critical response factor. If a powder coating provider makes a claim about a particular performance characteristic, the provider of the powder coating will be asked to provide a confidence limit and specification limit (acceptable quality limit) for that claim for verification purposes. If the provider does not provide a confidence and specification limit, a default of 95% confidence limit will be applied to all comparisons made to the target claim for the verification report.

If a claim about a particular performance characteristic is made by the powder coating provider, this claim will be used in the design of experiments to determine the appropriate number of panels to be coated and analyzed based on the confidence limit, specification limit, and the appropriate statistical test to be applied to the results (i.e., Student's t-Test, Chi Square Test, or F-Test). If there are no specific claims made by the powder coatings provider, then the default test will be comprised of five (5) separate runs with a maximum of four (4) standard products (2 products in each configuration) and 16 panels coated per run. This will enable total variation to be determined for each response factor with a reasonable statistical significance. In addition to these five (5) runs, a run using the standard powder coating will be done immediately before the five (5) runs. Data from the standard powder runs will be compared to historical data to assure the quality of the operation of the spraying equipment. The statistical analyses for all response factors will be carried out using the latest version of Minitab statistical software.

The powder coating to be tested will be applied using the standard apparatus set-up given above and the powder coating manufacturer's recommendations. Note that the powder coating of panels provides data for comparison with industry standards, as panels are typically used by coatings and equipment manufacturers to verify performance.

The test specimens will be hung on the conveyor and coated while passing in front of the spray equipment at a standard line speed recommended for the particular powder coating and spray equipment used. A run will consist of a maximum of the following:

- Two (2) standard test products hung with the outer surfaces facing the spray equipment
- Two (2) standard test products hung with the inner surfaces facing the spray equipment
- Two (2) racks of eight (8) standard test panels
- Two (2) test parts in any configuration specially requested to be coated by the technology provider in response to the RFT (test parts to be provided by the technology provider).

As for the VOC and HAP content of the powder coating under curing conditions, five (5) powder samples will be taken for VOC content determination and five (5) samples of the powder will be taken for HAP determination.

2.2.7 Performance Testing

CTC will provide the powder coatings providers with key non-critical factors to be used for testing, such as the standard apparatus and set-up. The powder coating providers will supply *CTC* with all appropriate spray equipment settings whenever applicable. The powder coating providers will be afforded the opportunity to assist *CTC* personnel during the start-up phase of the coating process.

Performance tests will be used to measure powder performance when coating standard test panels and products. A number of laboratory test procedures will be used to analyze the powder coating. These procedures will include both quantitative and qualitative measurements (see Table 5).

2.2.8 Participation

The Demonstration Factory at *CTC* provides a unique capability for demonstrating and evaluating full-scale manufacturing process applications. Production-scale processing and testing can be carried out on any of the process technologies within the Demonstration Factory without concern for the many problems associated with trying to do these same tests on in-service production processes. Because of this existing capability, most of the tests and demonstrations will be performed at the Demonstration Factory. The *CTC* technical staff will be responsible for performing all necessary tests and demonstrations required for performance evaluation and full-scale validation. Where specific equipment is required for testing and is not available, *CTC* will work with other facilities to perform the required work.

Providers of the powder coatings being tested will also be invited to participate in the testing. Their participation will ensure proper coating usage.

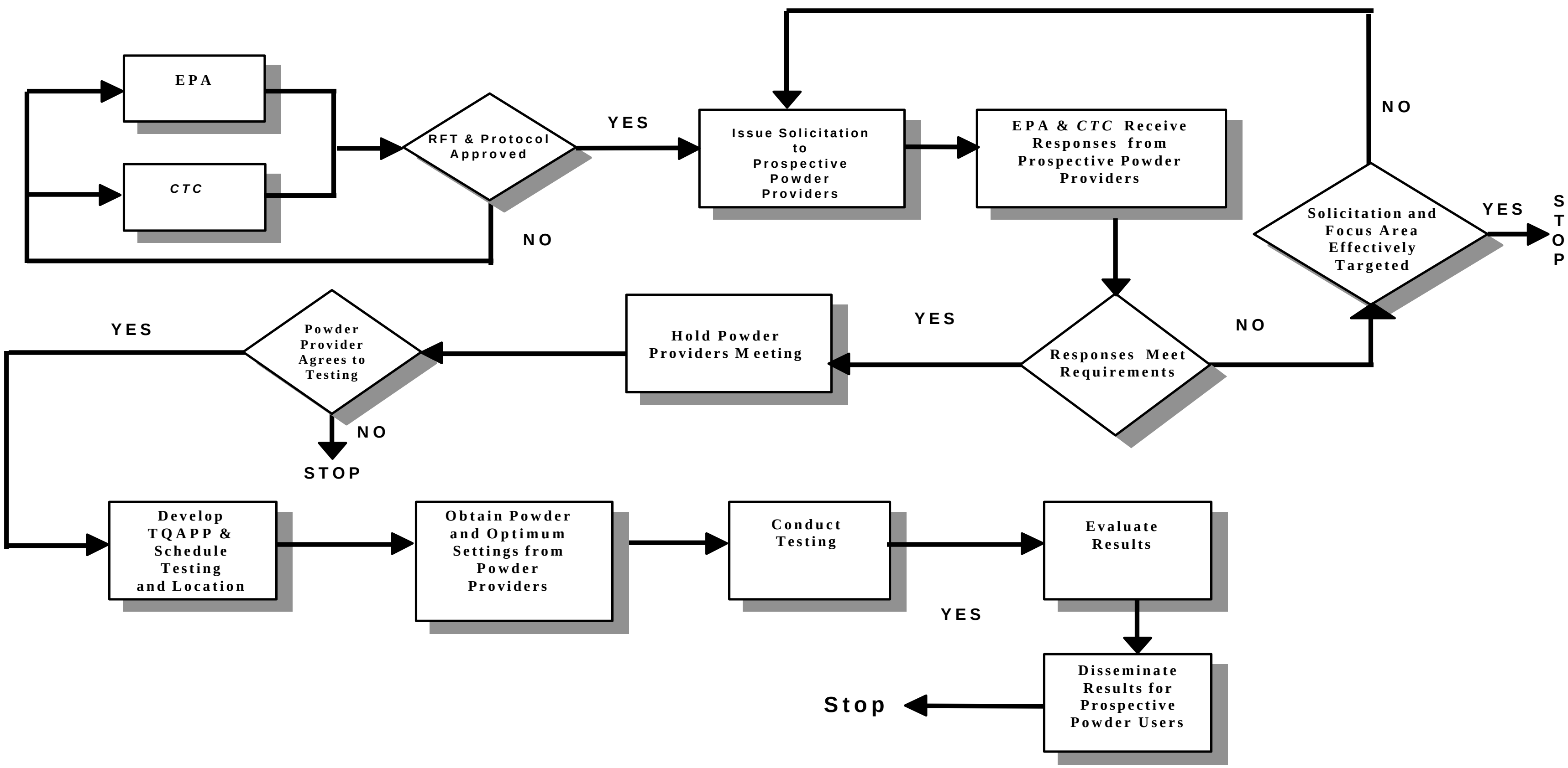

Figure 4. Schematic Diagram of Testing Program

2.2.9 Critical and Non-Critical Factors

For the purposes of this protocol, the following definitions will be used for critical control factors, non-critical control factors, and critical response factors. A critical control factor is a factor which is varied in a controlled manner within the design of experiments matrix to determine its effect on a particular outcome of a system. Non-critical control factors are all the factors which are to be held constant (or relatively so) or randomized throughout the testing. Critical response factors are the measured outcomes of each combination of critical control factors given in the design of experiments.

In this context the term “critical” does not convey the importance of a particular factor (that can only be determined through experimentation and characterization of the total process), but its relationship within the design of experiments. In the case of verification testing of a particular powder coating, there is only one critical control factor, and that is the powder coating itself. All other processing factors will be held constant (or randomized) and are non-critical control factors. Therefore, the multiple runs and sample measurements within each run for each critical response factor will go to determine the amount of variation expected for each critical response factor.

For all projects, the critical control factors, non-critical control factors, and critical response factors will be identified in a table format along with acceptance criteria (where appropriate), data quality indicators, measurement locations, and measurement frequencies broken down by each trial or experiment. For example, for a new low emission powder coating, parameters associated with metal surface pretreatment would remain constant and thus be non-critical control factors, while parameters such as coating performance and VOC content would be identified as critical response factors.

The only critical control factor (see Table 3) is the actual powder coating. The powder coating provider’s recommendations for optimum usage of the coating will be followed. As a result, some non-critical control factors (see Table 4) will likely vary from one powder coating to another.

The critical response factors which will be measured during the testing are given in Table 5. These critical response factors will be used to determine the performance of the powder coating. Whenever possible, standard ASTM methods will be used to determine the critical response values (see Table 9).

Tables 3 through 5 below summarizes the critical and non-critical factors which will be monitored throughout the testing.

Table 3. Critical Control Factors

Critical Control Factor	Set Points/ Acceptance Criteria	Measurement Location	Frequency	Total Number
Powder	N/A	N/A	N/A	N/A

Table 4. Non-Critical Factors

Non-Critical Factor	Set Points/ Acceptance Criteria	Measurement Location	Frequency	Total Number
Delivery Pressure	Based on Equipment Used	Based on Equipment Used	Continuous	N/A
Utility Needs	Based on Equipment Used	Factory Gauges Based on Equipment Used	Continuous	N/A
Zinc Phosphate Pretreatment Weight	Constant Zinc Phosphate weight per unit area	Random panel selected prior to the spray booth. Actual weight measurement per ASTM B 767	2 Panels selected: 1 Panel (randomly selected) per Rack, 2 Racks per run	10
Gel Time	From Powder Coating Provider	From ASTM D 3451	5 Samples from powder coating lot to be used during test	5
Inclined Plate Flow	From Powder Coating Provider	From ASTM D 3451	5 Samples from powder coating lot to be used during test	5
Specific Gravity	From Powder Coating Provider	From ASTM D 5965	1 Sample each run	5

Table 4. Non-Critical Factors (continued)

Non-Critical Factor	Set Points/ Acceptance Criteria	Measurement Location	Frequency	Total Number
Products involved in the testing	Panels and Standard Test Product (see Appendix A)	N/A	4 Standard Products and 16 Panels per Run	20 Standard Products and 80 Panels
Total Surface Area to be Coated	17.33 ft^2/run	Top and right edge of panels and exterior edges of standard test products	1 Test panel and 1 standard test product per test	2
Ambient Factory Temperature	70 - 80°F	Factory Floor	Continuous	N/A
Ambient Factory Relative Humidity	< 60% RH	Factory Floor	Continuous	N/A
Curing Oven Temperature	From Powder Coating Provider	Programmable Logic Controller (PLC)	Continuous	N/A
Curing Time	From Powder Coating Provider	Factory floor	Once each run	5
Spray Booth Air Flow	Designed for approx. 11,000 cfm	Factory floor	Once per test	1

Qualitative non-critical control factors used in this protocol include:

- Equipment Preparation — from powder coating provider
- Utility Requirements — from spraying equipment vendor
- Throughput — from powder coating provider
- Target Dry Film Thickness — 1.0 – 1.25 mil.
- Particle Size Distribution — from powder coating provider
- Curing Schedule — from powder coating provider

Table 5. Critical Response Factors*

Critical Response Factor	Measurement Location	Frequency	Total Number
Environmental			
Volatile Matter Content of Powder	Method Under Development[1]	5 Samples from powder coating lot to be used during test	5
Hazardous Air Pollutant Content of Powder	Method Under Development[1]	5 Samples from powder coating lot to be used during test	5
Energy Usage	Factory	Each Run	5
Durability			
Salt Spray	from ASTM B 117	5 Randomly Selected Panels per Run, 1 test per Panel	25
Adhesion	from ASTM D 3359	5 Randomly Selected Panels per Run	25
Impact	from ASTM D 2794	5 Randomly Selected Panels per Run	25
Flexibility (Mandrel Bend)	from ASTM D 522	5 Randomly Selected Panels per Run, 1 test per Panel	25
Pencil Hardness	from ASTM D 3363	5 Randomly Selected Panels per Run, 1 test per Panel	25
Pencil Hardness	from ASTM D 3363	5 Randomly Selected Panels per Run, 1 test per Panel	25
MEK Rub	from ASTM D 4752	5 Randomly Selected Panels per	25

Table 5. Critical Response Factors*

Critical Response Factor	Measurement Location	Frequency	Total Number
		Run, 1 test per Panel	

Table 5. Critical Response Factors (continued)*

Critical Response Factor	Measurement Location	Frequency	Total Number
Humidity Resistance	From ASTM D 1735	1 Sample per run	5
Weather Resistance	From ASTM G 26	1 Sample per run	5
Abrasion Resistance	From ASTM D 4060	1 sample per run	5
Other			
Overall Film Thickness (Magnetic method)	9 points in a 3 x 3 array on each coated face of the Standard Product	27 points per 5 Standard Products per 2 Orientations per Run	1350
Comparison of Edge Film Thickness to Non-Edge Film Thickness (Magnetic method)	3 points on each face farthest from the shared corner of the Standard Product compared to the rest of the points measured for the Overall Film Thickness above	27 points per 5 Standard Products per 2 Orientations per Run (same points as above)	1350 (same points as above)
Gloss	from ASTM D 523	5 Randomly Selected Panels per Run, 1 test per Panel	25
Color	from ASTM D 1729	5 Randomly Selected Panels per Run, 1 test per Panel	25
Color	from ASTM D 2244	5 Randomly Selected Panels per Run, 1 test per Panel	25

1 The US EPA is currently developing a test method for the measurement of VOCs and HAPs from powder coatings under curing conditions (time and temperature).

* See Section 2.2

2.3 Schedule

CTC uses standard tools for project scheduling. Project schedules are prepared in Microsoft Project or Primavera formats which are accepted industry standards for scheduling. Project schedules show the complete work breakdown structure (WBS) of the project, including technical work, meetings and deliverables. The estimated (planned) schedule for the various project activities is shown in Table 6.

Table 6. Estimated Project Schedule

ID	Name	Duration	Start Date	Finish Date
Task 1	Conduct initial stakeholders meeting	1d	03/21/97	3/21/97
Task 2	Investigate/identify/ prioritize focus areas	60d	3/21/97	5/16/97
Task 3	Draft and revise CBD/RFT for powder coatings	14d	5/16/97	7/1/97
Task 4	Approval and issuance of CBD/RFT	1d	7/1/97	7/15/97 (CBD) 7/30/97 (RFT)
Task 5	Draft and revise Generic Testing and Quality Assurance Protocol for powder coatings	45d	7/15/97	10/24/97
Task 6	Receive/review CBD/RFT responses	20d	Open	Open
Task 7	Approval and issuance of final Generic Testing and Quality Assurance Protocol	14d	Open	Open
Task 8	Stakeholder conference call to choose pertinent CBD/RFT responses for verification testing	1d	Open	Open
Task 9	Produce & obtain approval for specific TQAPPs for each powder coating to be tested	10d each	Open	Open
Task 10	Verification testing	Dependent on each powder coating	Open	Open
Task 11	Prepare test report	20d	Open	Open

Table 6. Estimated Project Schedule (continued)

ID	Name	Duration	Start Date	Finish Date
Task 12	Approval of test report by EPA	30d	Open	Open
Task 13	Verification statement - Issued by EPA	60d	Open	Open

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

CTC employs a matrix organization, with program and line management, to perform projects. The laboratory supports Project Managers and Technical Project Leaders by providing testing data. Laboratory Analysts report to the Laboratory Manager. The Laboratory Manager coordinates with the Technical Project Manager on testing schedules. The Technical Project Leader answers directly to the Project Manager of a task. The Technical Project Leader is the conduit between the laboratory and the Project Manager. Additionally, a Quality Assurance (QA) Engineer, who is independent of both the laboratory and the program or project, is responsible for developing and administering Division policies. These policies provide for, and ensure that quality objectives are met for each project, and cover laboratory testing, factory demonstration processing, engineering decisions, and deliverables. The QA Engineer reports directly to *CTC* senior management and is organizationally independent of project or program management.

The project organization chart, showing lines of responsibility and the specific *CTC* personnel assigned to this project, is presented in Figure 5. A summary of the responsibilities of each *CTC* participant, their applicable experience, and their anticipated time dedication to the project during testing and reporting is given in Table 7.

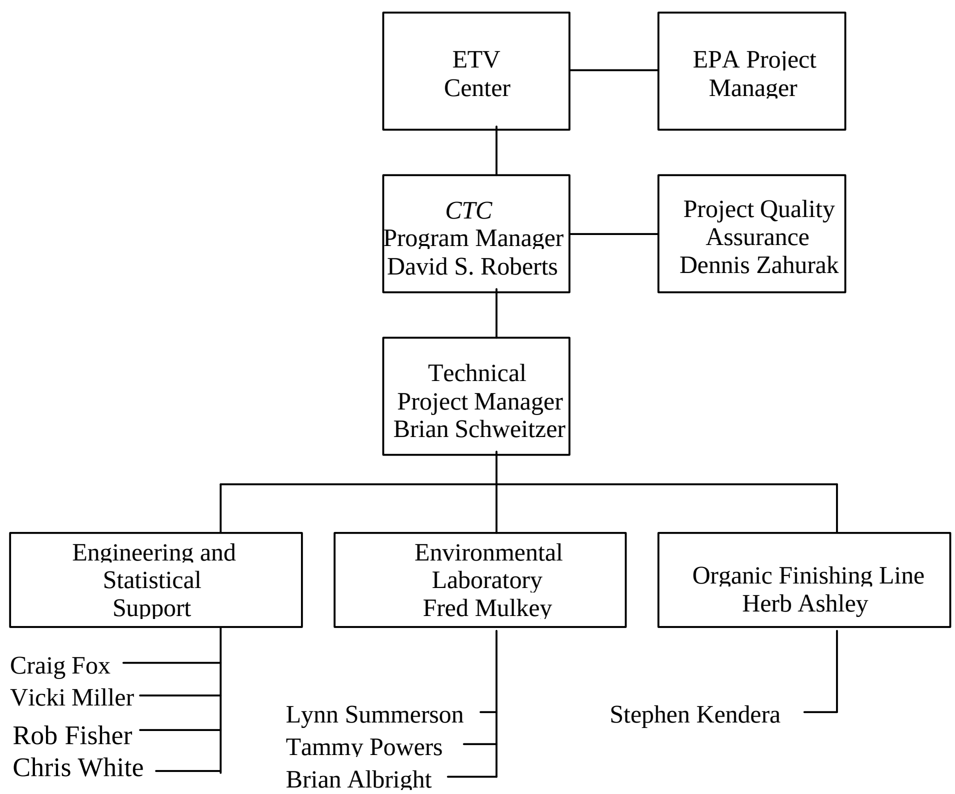

Figure 5. Project Organization Chart

Table 7. Summary of ETV CCEP Experience and Responsibilities

Key *CTC* Personnel and Roles	Responsibilities	Applicable Experience	Education	Time Dedication
Dave Roberts NDCEE Program Manager	Directs NDCEE Program. Accountable to *CTC* Technical Services Director and *CTC* Corporate Management.		B.S. Mechanical Engineering	5%
Brian Schweitzer Technical Project Manager	Responsible for overall ETV CCEP technical, budget, and schedule issues on daily basis. Accountable to NDCEE Program Manager & EPA.	Process Engineer (9 years) Project Manager, Organic Finishing (4 years)	B.S. Mechanical Engineering	50%
Craig Fox Sr. Engineer	Technical Project Support. Project Management Support. Design of Experiments. Accountable to Project Manager.	Industrial Process R&D (10 years) Project Management (10 years) Industrial Design of Experiments (8 years)	M.S. Chemical Engineering, B.S. Chemical Engineering	50%
Vicki Miller Associate Process Engineer	Technical project support. Process design & development. Accountable to Project Manager.	Associate Process Engineer, Organic Finishing (2 years)	B.S. Chemical Engineering	75%

Table 7. Summary of ETV CCEP Experience and Responsibilities (continued)

Key *CTC* Personnel and Roles	Responsibilities	Applicable Experience	Education	Time Dedication
Chris White Associate Process Engineer/ Technical Project Leader	Technical project support. Process design & development. Accountable to Project Manager.	Associate Process Engineer (4 years)	B.S. Chemical Engineering	40%
Rob Fisher Staff Process Engineer/ Technical Project Leader	Technical project support. Process design & development. Accountable to Project Manager.	Organic Finishing Regulations (5 years)	B.S. Chemical Engineering	40%
TBD Quality Assurance	Responsible for overall project QA.			5%
Herb Ashley Finishing Engineer Factory Operations Lead	Oversees day-to-day operation of Organic Finishing Line. Provides technical project support. Accountable to Project Manager.	Organic Finishing Experience (26 years)		10%

Table 7. Summary of ETV CCEP Experience and Responsibilities (continued)

Key *CTC* Personnel and Roles	**Responsibilities**	**Applicable Experience**	**Education**	**Time Dedication**
Stephen Kendera Sr. Organic Finishing Technician	Performs day-to-day operations of the Organic Finishing Line. Accountable to Finishing Engineer.	Industrial Paint and Coatings Experience (25 years)		10%
Fred Mulkey Manager, Laboratory Operations	Project TQAPPs. Coordinates Testing Lab; Technical data review. Accountable to Project Manager, NDCEE Program Manager.	Laboratory Chemist and Manager Project Quality Assurance Project Management (10 years)	M.S. Chemistry, B.S. Chemistry	5%
Tammy Powers Associate Laboratory Leader	Laboratory analysis. Accountable to Laboratory Manager.	Environmental and Municipal Laboratory Testing (7 years)	B.S. Biology	10%
Lynn Summerson Laboratory Leader	Laboratory analysis. Accountable to Laboratory Manager.	Industrial and Environmental Laboratory Testing (17 years)	M.S. Chemistry	20%
Brian Albright Assistant Laboratory Analyst Pretreatment Operator	QC Analysis. Accountable to Laboratory Manager.	Environmental and QC Testing (3 years)	B.S. Chemistry	10%

Table 7. Summary of ETV CCEP Experience and Responsibilities (continued)

Key *CTC* Personnel and Roles	Responsibilities	Applicable Experience	Education	Time Dedication
Carl Izzo Independent Industrial Paint Consultant	Technical project support. Process design & development. Accountable to Project Manager.	Industrial Coatings Research, Development, and Applications (40+ years)	B. S. Chemistry	Consultant

The *CTC* personnel specified in Figure 5 and Table 7 are responsible for maintaining communication with other responsible parties working on the project. The frequency and mechanisms for communication are shown in Table 8.

Table 8. Frequency and Mechanisms of Communications

Initiator	Recipient	Mechanism	Frequency
Project Manager or Technical Project Leader	EPA Project Manager	Written Report	Monthly
Technical Project Manager	Program Manager	Written or Verbal Status Report	Weekly
Laboratory Manager	Technical Project Manager	Data Reports	As Generated
QA Engineer	Program Manager	Quality Review Report	As Required
EPA Project Manager	*CTC*	On-Site Visit	At Least Once per year

Table 8. Frequency and Mechanisms of Communications (continued)

Special Occurrence	Initiator	Recipient	Mechanism/ Frequency
Schedule or Financial Variances	Program Manager or Technical Project Manager	EPA Project Manager	Telephone Call, Written Follow-up Report as Necessary
Major (will prevent accomplishment of verification cycle testing) Quality Objective Deviation	Program Manager or Technical Project Manager	EPA Project Manager	Telephone Call with Written Follow-up Report

4.0 PROJECT QUALITY ASSURANCE (QA) OBJECTIVES

4.1 General Objectives

The overall objective of the ETV CCEP is to verify the pollution prevention characteristics of powder coatings, and to make the results of the verification testing available to prospective powder coatings users. This objective will be met by controlling and monitoring the critical and non-critical factors, which are the specific QA objectives for this protocol. Critical and non-critical indicators will be established with the data source arising from one or more of the following categories:

- the powder coating process (chemical control as well as technique)
- raw materials, including powder coatings
- equipment, components, sensors
- product quality
- multi-media environmental aspects
- health and safety
- life-cycle costs (capital investment, utilities, labor, waste handling, operation and maintenance, and others).

The analytical methods that will be used for powder coating evaluation are adapted from ASTM Standards. The QA objectives of the project and the capabilities of these test methods for product and process inspection and evaluation are synonymous since the methods were specifically designed for evaluation of the powder coating properties under investigation. The methods will be used as published, or as supplied, without major deviations. A list of the specific methods to be used for this project are attached to this document in Appendix E.

4.2 Quantitative Quality Assurance Objectives

Quality assurance (QA) objectives will be established for all critical measurements and for each sample type. Both physical and chemical measurements will be considered in the evaluation and establishment of critical and non-critical measurements. The QA objectives will be stated quantitatively (or qualitatively where appropriate) in such a manner to allow overall project objectives to be met. For example, if a rinse water discharge for a powder coating is being monitored for Clean Water Act compliance, then the QA objectives and test methods will be set to allow measurement at appropriate detection levels and confidence intervals for the data.

It is anticipated that the measurement systems required for ETV CCEP projects primarily will be standard technologies rather than research measurement tools, and the QA objectives will be achieved using nationally accepted testing and calibration methodologies. Any nonstandard methods are fully documented with supporting data provided in the appendix to the TQAPP. Establishing Quality Assurance objectives will be a collaborative effort involving laboratory, engineering, quality assurance and project management personnel to ensure reasonableness and validity of these objectives. Quality assurance objectives will be clearly stated as well as compiled in tables.

The statistical support engineer, quality assurance engineer, and laboratory personnel will coordinate efforts to determine the manner in which test results and QA objectives will be interpreted in a statistical sense.

4.2.1 Accuracy

Standard reference materials, traceable to national sources such as the National Institute for Standards and Technology (NIST) for instrument calibration and periodic calibration verification, will be procured and utilized where such materials are available and applicable to this project. For reference, calibration materials with certified values, acceptable accuracy for calibration verification will be 80-120 percent of the true reference values, or within the method specific guidelines when given. Reference materials will be evaluated using the same methods as for the actual test specimens.

4.2.2 Precision

The experimental approach of this project specifies the exact number of panels and standard test products to be coated. The analysis of replicate panels for each powder coating property at each of the experimental conditions will occur by design. The degree of precision will be assessed based on the agreement of all replicates within a property test group.

4.2.3 Completeness

The laboratory strives for 90 percent completeness. Completeness is defined as the number of valid determinations expressed as a percent of the total number tests conducted, by test type.

4.2.4 Impact and Statistical Significance of Quality Objectives

Data from the product analyses should meet the accuracy and completeness requirements specified in Table 9 below. The precision requirements also should be achieved; however, a non-conformance may result from the analysis of replicates due to limitations of the powder coating technology under evaluation, and not due to processing equipment or laboratory error. Regardless, if any non-conformance from QA objectives occurs, the cause of the deviation will be determined by checking calculations, verifying the testing and measuring equipment, and re-analysis. If an error in processing is discovered, re-processing of a new batch for a given trial will be considered and the impact to overall project objectives determined. If the deviation persists despite all corrective action steps, the data will be flagged as not meeting the specific quality criteria and a written discussion will be generated.

If all process conditions are within control limits and instrument and/or measurement system accuracy checks are valid, the nature of the non-conformance may be beyond the control of the laboratory. Given that laboratory quality control data are within specification, these results will be interpreted as the inability of a particular powder coating undergoing testing to produce parts meeting claimed performance criteria for the powder coating at a given set of experimental conditions, if a claim about a particular performance characteristic has been made.

4.3 Qualitative QA Objectives: Comparability and Representativeness

4.3.1 Comparability

Powder coatings will be utilized and/or operated at vendor/supplier recommended conditions or conditions otherwise established in agreement with project stakeholders. The data will be comparable from the standpoint that other testing programs could reproduce similar results using the same powder coatings and documented process instructions. Powder coating and environmental performance will be evaluated using EPA, ASTM and other nationally or industry wide accepted testing procedures. Process performance parameters and cost data will be generated and evaluated according to standard best engineering practices. In addition, suppliers will be asked to provide performance data for their product and the results of preliminary or prior testing as relevant to the specific project, if available.

Table 9. QA Objectives for Precision, Accuracy and Completeness for Sample Powder Coating Analyses

Measurement	Method	Units	Precision	Accuracy	Completeness
Salt Spray	ASTM B 117	Pass/Fail	All Pass or All Fail	N/A	N/A
Film Thickness--Magnetic	ASTM B 499	mils	20%	10% True Thickness	90%
Zinc Phosphate Pretreatment Weight	ASTM B 767	g/m^2	$\pm$0.005 g/m^2	$\pm$0.01 g/m^2	90%
Total Surface Area to be Coated	Caliper	In^2/panel	$\pm$0.08 in^2/panel	$\pm$0.1%	90%
Ambient Factory Temperature	Thermal Hygrometer	^oC	$\pm$3% of full scale	$\pm$3% of full scale	90%
Ambient Factory Relative Humidity	Thermal Hygrometer	%RH	$\pm$3% of full scale	$\pm$3% of full scale	90%
Curing Oven Temperature	Thermocouple/ (controllers)	^oC	$\pm$2.2^oC	$\pm$2.2^oC	90%
Curing Time	Stopwatch	min	$\pm$0.001%	$\pm$0.001%	90%
Spray Booth Air Flow	Per ACGIH	ft/min	(2)	(2)	(2)
Adhesion by Tape Test	ASTM D 3359	Pass/Fail and the 0-5 Rating	All Pass or All Fail	$\pm$ 1 Rating	N/A
Impact (Direct & Reverse)	ASTM D 2794	Pass/Fail	All Pass or All Fail	See ASTM D 2794 for ranges	N/A
Flexibility (Mandrel Bend)	ASTM D 522	Pass/Fail	All Pass or All Fail	$\pm$ 15%	N/A
Pencil Hardness	ASTM D 3363	Hardness Scale	One Pencil Unit	$\pm$ 1 pencil unit	90%
MEK Rub	ASTM D 4752	0-5 Rating	One Rating Unit	N/A	90%
Gloss	ASTM D 523	gloss units	20%	See ASTM D 523 for ranges	90%
Color--SpectraLight II	ASTM D 1729	Visual	N/A	N/A	N/A
Color--Spectrometer	ASTM D 2244	ΔE Values	20%	$\pm$ 0.2 ΔE Values	90%
Humidity Resistance	ASTM D 1735	Pass/Fail	All Pass or All Fail	N/A	N/A

Table 9. QA Objectives for Precision, Accuracy and Completeness for Sample Powder Coating Analyses (continued)

Measurement	Method	Units	Precision	Accuracy	Completeness
Weather Resistance	ASTM G 26	Pass/Fail	All Pass or All Fail	N/A	N/A
Abrasion Resistance	ASTM D 4060	Milligrams	46%	Not reported in ASTM D 4060	90%
VOC Content of Powder[1]	TBD	TBD	TBD	TBD	TBD
HAP Content of Powder[1]	TBD	TBD	TBD	TBD	TBD
Gel Time	ASTM D 3451	Seconds	20%	Not reported in ASTM D 3451	90%
Inclined Plate Flow	ASTM D 3451	Millimeters	20%	Not reported in ASTM D 3451	90%
Specific Gravity	ASTM D 5965	g/cc	$\pm$0.04 g/cc	Not determined	90%

1 The US EPA is currently developing a test method for the measurement of VOCs and HAPs from powder coatings under curing conditions (time and temperature).

2 Accuracy and Precision stated by the manufacturer as 100-600 ft/min: $\pm$3% of reading $\pm$2 ft/min

Test specimens generated at *CTC* will be compared to these performance data and to the other applicable end user and industry specifications. These performance standards will be used to indicate if the technology under consideration meets project objectives. Additional assurance of comparability comes from the routine use of precision and accuracy indicators, as described above; the use of standardized and accepted methods; and traceability of reference materials.

4.3.2 Representativeness

The limiting factor to representativeness is the availability of a large sample population. Experimental designs will be constructed such that projects will have either sufficiently large sample populations per trial or otherwise statistically significant fractional populations. The trials will be conducted at the paint and equipment supplier-recommended operating conditions. If the test data meets the quantitative QA criteria (precision, accuracy, and completeness) then the samples will be considered representative of the powder coating technologies under evaluation and will be used for interpreting the outcomes relative to the specific project objectives.

4.4 Other QA Objectives

In addition to primary data QA objectives, individual projects may require additional QA objectives for mass balancing, health and safety, economic factors, or life-cycle assessment. When these objectives are part of a project, these will be stated in quantitative and qualitative terms as appropriate.

For example, a mass balance may be required to account for the total amount of material used in a powder coating operation. This would require a series of direct and indirect indicators such as gravimetric and specific test procedures as well as the calculation or approximation of materials where direct measurement is not possible, feasible, or cost effective. Another example is the calculation of cost factors for implementation of a technology where scale-up factors to production level and other engineering estimates must be used. The exact approaches taken will be specified in each TQAPP.

4.5 Impact of Quality

Due to the highly controllable nature of the test panel evaluation methods and predictability of factors affecting the quality of the laboratory testing of panels, the quality control of test panel qualifications is expected to fall within acceptable levels. Deviation from quantitative and qualitative QA objectives is not expected.

5.0 SITE SELECTION AND SAMPLE HANDLING PROCEDURES

5.1 Site Selection

This project will be executed at *CTC*, and whenever possible all processing and testing will be performed by *CTC* personnel. The site for application and evaluation will be at *CTC* in the Demonstration Factory at the Environmental Technology Facility (ETF) under the direct control of the Engineering and Statistical Support and Organic Finishing Line Groups. Analysis will be performed in the *CTC* Testing Laboratory at the ETF by the Environmental Laboratory. The application of the powder coating involves transporting test panels or parts via automatic conveyor through the organic finishing line. The panels will be pretreated within the seven-stage pretreatment process in the organic finishing line and then coated in the powder coat subsystem. Test panels will be evaluated after curing and cooling.

The experimental design involves applying a powder coating according to the suppliers recommended conditions. The panels or parts will be sampled and analyzed to generate performance data. Control batches may be tested for the same properties for comparative data.

5.2 Site Description

Please refer to Figure 2 for an overall layout in the Demonstration Factory of the process equipment that may be used for the evaluation of the powder coating technologies. The testing to be completed for this project involves the use of the pretreatment process, the dry-off oven, the powdercoat subsystem, and the powder curing oven shown in Figure 3. Other equipment or testing sites may be utilized as necessary.

5.3 Sampling Procedures and Handling

Test panels and standard test products, along with any technology provider specified test products (if any), will be used in this project. These will be pre-labeled by stamping with a unique alpha-numeric identifier. The number of specimens processed during the testing depends upon the experimental design, which in turn depends on any provider's claim(s) about a particular performance characteristic(s). Unless all of the powder coating performance characteristics require a lesser number of samples, the default experimental design will be used. This experimental design is based on a maximum number of 10 samples (2 from each of 5 runs) per critical response factor. The default experimental design is outlined in more detail in Section 2.2.5.

A factory operations technician will process the panels and standard test products according to a pre-planned sequence of stages, including: application of the powder coating to the panel, curing, and cooling. A laboratory analyst will take possession of the samples from the factory personnel, and process the samples through the laboratory sample login station. The date and time of processing and the process conditions will be recorded for each trial. Samples taken to the laboratory for analysis will be labeled with the date/time sampled, the initials of sampling personnel, and they will be given a unique laboratory identification number.

When selecting a sampling site, consideration will be given to the following as specified in the experimental design:

- population size and reason for selection
- description of sample type (whether panels, parts, wastes, etc.)
- type of sampling strategy (whether simple, stratified, etc.)
- statistical methods used and rationale
- frequency and number of samples taken
- sources of contamination
- effects of site selection on data validity.

5.4 Sample Custody, Storage and Identification

The test panels will be delivered to the laboratory sample login station. The analyst delivering the panels will complete a custody log indicating the sampling point ID's, sample material ID's, quantity of samples, time, date and analyst's initials. The product evaluation tests also will be noted on the custody log. The laboratory's sample custodian will verify this information. Both personnel will sign the custody log to indicate transfer of the samples from the powder coating processing area to the laboratory analysis area. The laboratory sample custodian will log the panels into a bound record book; store the panels under appropriate conditions (ambient room temperature and humidity); and create a work order for the various laboratory departments to initiate testing. Testing will begin within several days of powder coating application.

6.0 ANALYTICAL PROCEDURES AND CALIBRATIONS

6.1 Facility and Laboratory Testing and Calibration

CTC has developed and maintains a calibration system within both the factory and the laboratory. Testing and measuring equipment are calibrated on a periodic basis to ensure that the data collected are accurate.

6.1.1 Facility Testing and Calibration

Calibration procedures within the factory are derived from ISO 10012-1 and MIL SPEC 45662A guidelines. A software package is used to track calibration information for each piece of testing and measuring equipment. This software serves to alert personnel when each piece of equipment is scheduled for calibration. Certified solutions and reference materials traceable to National Institute of Standards and Technology (NIST) are purchased when they are available. Where a suitable source of material does not exist, a secondary standard is prepared and a true value obtained by measurement against a NIST traceable standard.

6.1.2 Laboratory Testing and Calibration Procedures

The analytical methods performed at *CTC* are adapted from standard ASTM, MIL-SPEC, EPA, Association of Official Analytical Chemists (AOAC) and/or industry protocols for similar manufacturing operations. Initial calibration and periodic calibration verification are performed at the frequencies specified by the methodology to ensure that an instrument is operating sufficiently to meet sensitivity and selectivity requirements. At a minimum, all equipment is calibrated before use and is verified during use and/or immediately after each sample batch. Standard solutions are purchased from reputable chemical supply houses in neat and diluted forms. When available, the laboratory purchases reference materials and solutions that are certified and traceable to the National Institute of Standards and Technology (NIST) for calibration and standardization. Data from all equipment calibrations and chemical standard certificates from vendors are stored in laboratory files and are readily retrievable. Each calibration procedure is documented in a formal laboratory standard operating procedure for which the analyst conducting experiments is trained. The analyst is also trained to detect non-conforming calibrations from method specific QA checks. No samples are reported in which the full calibration curve or the periodic calibration check standards are outside method performance standards.

6.2 Product Quality Procedures

Apparatus used to assess the quality of a powder coating on a test panel is set-up and maintained according to the manufacturer's and the published reference method's instructions. Actual sample analysis will take place only after set-up is verified per the reference method and the powder coating manufacturer's instructions. As available, samples of known materials with established product qualities are used to verify that a system is functioning properly. For example, traceable thickness standards are used to calibrate the eddy current thickness instrument. The remaining product quality tests that will be performed include adhesion, resistance to corrosion, visual appearance. Adhesion is a qualitative test for which calibration is not relevant. The scribes and other tools, including adhesive tape, used to destructively remove coating are checked for general condition and/or expiration. Corrosion resistance is another qualitative test for which calibration, per se, is not relevant. There are several equipment checks which are performed to ensure proper functioning of the salt spray chamber. These involve analysis of the solution (salt fog) collected from the chamber per the published method for collection rate, pH, and specific gravity (measure of salinity). Bare steel panels are also placed into the chamber and analyzed to ensure that the chamber is not excessively corrosive per ASTM requirements. Acids are made fresh for each test and weighing is performed on calibrated (traceable) balances. A list of applicable ASTM methods are attached as Appendix E.

6.3 Work Instructions (Standard Operating Instructions) and Calibration

Table 10 summarizes the methods and calibration criteria that will be used for the evaluation of the powder coatings. The laboratory creates a standard operating procedure (SOP) for each test that it performs on a routine basis adapted from published references, such as ASTM and EPA, and from accepted protocols provided by industrial suppliers. SOP’s are in the form of ISO 9000 Work Instructions. Work Instructions are created for equipment operation/sample analysis instructions, calibration and maintenance. The Laboratory Manager ensures that Work Instructions are created, reviewed and followed by laboratory personnel. The Work Instructions adhere to the quality elements contained in the original reference sources. The format for a laboratory Work Instruction is as follows:

- Title, Controlled ID #, Revision #
- Purpose
- Applicability
- Summary of Method
- Definitions
- Supporting Documents
- Equipment and Materials
- Training
- Environment, Health and Safety
- Calibration and verification
- Maintenance
- Instruction/Process

6.4 Non-Standard Methods

For methods which are non-standard (i.e., no commonly accepted or specified method exists or no traceable calibration materials exist), procedures will be performed according to the manufacturer's instructions or to the best capabilities of the equipment and the laboratory. This information will be documented in an SOP format. The performance will be judged based on the manufacturer’s specifications, or will be judged based on in-house developed protocols. These protocols will be similar or representative in magnitude and scope to related methods performed in the laboratory, which do have reference performance criteria for precision and accuracy. For instance, if a non-standard quantitative chemical procedure is being performed, it should produce replicate results of +/- 25 relative percent difference and should give values within +/- 20 percent of true or expected values for calibration and percent recovery check samples. For qualitative procedures, replicate results should agree as to their final evaluations of quality or performance (i.e., both should either pass or both should fail if sampled together from a properly functioning process). The intended use and any limitations would be explained in a SOP for a non-standard procedure; however, for this project, *CTC* does not intend to use any non-standard methods.

Table 10. Product Evaluation Testing Procedures and Calibration Criteria

Critical Measurement	Method Number[1]	Method Type	Calibration Procedure	Calibration Frequency	Calibration Accept. Criteria[2]
Salt Spray	ASTM B 117	Salt Fog 5% NaCl Neutral pH	Verify collection rate, pH, salinity, and bare steel corrosion rate	Weekly chemical tests, monthly steel tests	20% Relative Standard Deviation (RSD) among steel panels, avg. of chemical tests within specific ranges
Film Thickness	ASTM B 499	Magnetic	Multi-point curve with NIST traceable standards	Each use, verify calibration after 10 samples	90-110%
Zinc Phosphate Pretreatment Weight	ASTM B 767	Chromate Solution 50g/L CrO3	Comparison to NIST traceable standard	With each use	80-120%
Total Surface Area to be Coated	Caliper	Caliper	Comparison to NIST traceable standard	Annually	$\pm$0.001 in
Ambient Factory Temperature	Thermal Hygrometer	Thermal Hygrometer	Return to manufacturer	Annually	N/A
Ambient Factory Relative Humidity	Thermal Hygrometer	Thermal Hygrometer	Return to manufacturer	Annually	N/A
Curing Oven Temperature	Thermocouple/ (controllers)	Thermocouple/ (controllers)	Comparison to NIST traceable standard	Annually/ (six months)	$\pm 2.2^{o}C/(\pm 0.8^{o}C)$
Curing Time	Stopwatch	Stopwatch	Return to manufacturer	Six months	N/A
Spray Booth Air Flow	Per ACGIH	Anonometer	Return to manufacturer	Annually	N/A

[1]Copies of ASTM methods are provided in the Appendix.
[2]As a percent recovery of a standard.

Table 10. Product Evaluation Testing Procedures and Calibration Criteria (continued)

Critical Measurement	Method Number[3]	Method Type	Calibration Procedure	Calibration Frequency	Calibration Accept. Criteria[4]
Adhesion	ASTM D 3359	Tape Test	Verify condition of scribes and freshness of adhesives	Each use	N/A
Impact (Direct & Reverse)	ASTM D 2794	2 lb weight	Verify weight of indentor, verify ruler	Yearly	80-120%
Flexibility (Mandrel Bend)	ASTM D 522	Conical Mandrel	Verify conical diameter	Yearly	80-120%
Pencil Hardness	ASTM D 3363	Pencil	Supplier graded lead (use same supplier)	Each use	N/A
MEK Rub	ASTM D 4752	MEK Saturated Cheesecloth	Reagent grade MEK	N/A	N/A
Gloss	ASTM D 523	Glossmeter	Multi-point curve with NIST traceable standards	Each use, verify calibration after 10 samples	90-110%
Color	ASTM D 1729	Visual	N/A	N/A	N/A
Color	ASTM D 2244	Spectrometer	Zero with white tile	Each use	N/A
Humidity Resistance	ASTM D 1735	100% Humidity using Fog App.	Collection rate, pH	Daily collection rate and pH	Must be within specified ranges
Weather Resistance	ASTM G 26	Xenon arc with and without humidity	Irradiance, temperature, black panel, wet & dry bulb, wattage, water quality	Weekly	Must be within specified ranges
Abrasion Resistance	ASTM D 4060	Taber Abraser	Verify load weights	Each use	95-105%
VOC Content of Powder[1]	TBD	TBD	TBD	TBD	TBD
HAP Content of Powder[1]	TBD	TBD	TBD	TBD	TBD
Gel Time	ASTM D 3451	Hot Plate	Verify surface temperature with pyrometer	Yearly	190□C +/ - 1□C
Inclined Plate Flow	ASTM D 3451	Oven	Verify thermocouple	6 months	+/- 4□F or +/- 0.75% of the reading
Specific Gravity	ASTM D 5965	Method A	Comparison to NIST traceable standard	6 months	±0.04 g/cc

1 The US EPA is currently developing a test method for the measurement of VOCs and HAPs from powder coatings under curing conditions (time and temperature).

[3]Copies of ASTM methods are provided in the Appendix.
[4]As a percent recovery of a standard.

7.0 DATA REDUCTION, VALIDATION, AND REPORTING

7.1 Raw Data Handling

Raw data will be generated and collected by the analysts at the bench and/or process level. Process data is recorded into a process log during factory operations. A QA Checklist is also completed to ensure that the appropriate parts, panels, samples, and operational conditions are used. Bench data will include original observations, printouts and readouts from equipment for sample, standard and reference QC analyses. Data will be collected both manually and electronically. At a minimum, the date, time, sample ID, instrument ID, analyst ID, raw signal or processed signal, and/or qualitative observations will be recorded. Comments to document unusual or non-standard observations also will be included on the forms as necessary. Raw data will be processed manually by the analyst, automatically by an electronic program, or electronically after being key-punched into a computer. The analyst will be responsible for scrutinizing the data according to specified precision, accuracy, and completeness policies. Raw data bench sheets, calculations and data summary sheets will be kept together for each sample batch. From the written standard operating procedure and the raw data bench files, the steps leading to a final result may be traced.

7.2 Preliminary Data Package Validation

The generating analyst will assemble a preliminary data package. This package will contain the QC and raw data results, calculations, electronic printouts, conclusions and laboratory sample tracking information. A second analyst will review the entire package and may also check sample and storage logs, standard logs, calibration logs, and other files, as necessary, to ensure that tracking, sample treatments and calculations are correct. After the package has been peer reviewed in this manner, a preliminary data report will be prepared. The entire package and final report will be submitted to the Laboratory Manager.

7.3 Final Data Validation

The Laboratory Manager shall be ultimately responsible for all final data released from the laboratory. The Laboratory Manager will review the final results for adequacy to project QA objectives. If the manager suspects an anomaly or non-concurrence with expected or historical performance values, with project QA objectives, or with method specific QA requirements of the laboratory SOP, he will initiate a second review of the raw data and query the generating and reviewing analysts about the non-conformance. Also, he will request specific corrective action. If suspicion about data validity still exists after internal review of laboratory records, the manager may authorize a re-analysis. If sufficient sample is not available for re-testing, a re-sampling will occur. If the sampling window has passed, or re-sampling is not possible, the Laboratory Manager will flag the data as suspect and notify the technical project leader. The Laboratory Manager will sign and date the final data package.

7.4 Data Reporting and Archival

A report signed and dated by the Laboratory Manager is submitted to the technical project leader, the QA Engineer, and other technical principals involved in the project. The technical project leader will decide on the appropriateness of the data and will make any interpretations with respect to project QA objectives. The final laboratory report will contain the lab sample ID, date reported, date analyzed, the analyst, the SOP used for each parameter, the process or sampling point identification, the final result and the units. The laboratory will retain the data packages indefinitely. The lead technical engineer or the project manager will forward the results and conclusions to EPA in their regular reports, after obtaining corporate approvals.

7.5 Verification Statements

After receiving and approving a report of results from testing from the lead technical engineer or the project manager, the EPA will disseminate a verification statement for each technology evaluated after the technology providers approval via an approved dissemination plan.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Guide Used for Internal Quality Program

CTC is currently establishing an ISO 9001 operating program for its labs and the Demonstration Factory. The laboratory is currently establishing a formal quality control program for its specific operations. The format for laboratory quality assurance/quality control (QA/QC) is being adapted from several sources as follows:

Table 11. *CTC* Laboratory QA/QC Format Sources

ISO Guide 25	ISO Laboratory Quality Programs
Critical Elements for Laboratories	Pennsylvania Department of Environmental Resources
EPA Test Methods	SW-846
EPA Test Methods	100-300 series of methods
Ratliff, Thomas A.	The Laboratory QA System

8.2 Types of QA Checks

The ETF laboratory at *CTC* follows published methodologies, wherever possible, for testing protocols. Laboratory methods are adapted from Federal Specifications, Military Specifications, ASTM Test Methods, and supplier instructions. The ETF laboratory adheres to the QA/QC requirements specified in these documents. In addition, where QA/QC criteria are not specified, or where the laboratory performs additional QA/QC activities, these protocols are explained in the laboratory's SOPs (Work Instructions). Each facility using a supplier's product implements their own level of QA/QC. *CTC*'s laboratory at ETF will perform the testing and QA/QC verification outlined in Table 9 (Precision, Accuracy, Completeness) and Table 10 (Calibration); therefore, these tables should be referred to for the method specific QA/QC that will be performed.

8.3 Basic QA Checks

The laboratory monitors its reagent de-ionized water to ensure it meets purity levels consistent with analytical methodologies. The filters are replaced quarterly before failures are encountered. Samples are not processed until the filters are replaced when failures do occur. The quality of the water is assessed with method reagent water blanks. Blank levels must not exceed minimum detection levels for a given parameter to be considered valid for laboratory use.

Thermometers are checked against National Bureau of Standards (NBS) certified thermometers at two temperatures. The laboratory uses thermometers to check the temperature of sample storage areas, ovens, hot plate operations, and certain liquid baths and documents these checks.

Balances are calibrated by an outside organization using standards traceable to NIST. *CTC* also performs in-house, periodic verifications with ASTM Class 1 weights. Records of this activity and certificates are kept by the ETF laboratory. The laboratory analyst also checks the balances prior to use with ASTM Class 1 weights.

Reagents purchased directly by the laboratory are American Chemical Society (ACS) grade or better. Reagents are not used beyond their certified expiration dates. Reagents are dated upon receipt and when first opened.

Laboratory waste is segregated, according to chemical classifications, in labeled containers to avoid cross-contamination of samples.

8.4 Specific Checks

CTC's ETF laboratory will analyze blanks, replicates on separate and on the same samples; perform initial and periodic calibration checks; and will check any referenced materials and equipment, as available and specified by the referenced methodology and/or the project-specific QA/QC objectives. Laboratory records are maintained with the sample data packages and/or in centralized files, as appropriate. To ensure comparability, the laboratory will carefully control process conditions and perform product evaluation tests consistently for each specimen. The specific QA checks listed in Tables 9 and 10 provide the necessary data to determine if process control and product testing objectives are being met. ASTM, Federal, and Military methods that are accepted in industry for product evaluations, and supplier-endorsed methods for process control, will be used for all critical measurements, thus satisfying the QA objective. A list of the published methods to be used are included in Appendix E.

9.0 PERFORMANCE AND SYSTEMS AUDITS

CTC has developed a system of internal and external audits to monitor both program and project performance. These include monthly managers meetings and reports, financial statements, EPA reviews and stakeholders meetings, and In Process Reviews. The ETF laboratory also analyzes performance evaluation samples in order to maintain PA Department of Environmental Protection Certification.

ISO Internal Audits
CTC is establishing its quality system based on ISO 9000 and 14000 and will be implementing a system of ISO internal audits. This information will be used for internal purposes.

On-Site Visits
The EPA Project Officer may visit *CTC* for an on-site visit during the execution of this project. All project, process, quality assurance, and laboratory testing information will be available for review.

EPA Audits
The EPA will periodically audit *CTC* during the execution of projects. All project, process, quality assurance, and laboratory testing information will be made available per the EPA's auditing procedures.

Technical Systems Audits
A listing of all powder coating equipment, laboratory measuring and testing devices, and procedures, powder coating procedures, and a copy of the final approved QA plan will be given to the project QA engineer. The QA engineer will conduct an initial audit, and audits thereafter of production and testing activities. The results of this activity will be forwarded to EPA in quarterly reports from the Program Manager or the technical project leader.

Audits of Data Quality
Peer review in the laboratory constitutes a process whereby raw data generated at the bench level are reviewed by two analysts. After data are reduced they undergo review by laboratory management. For this project, laboratory management will spot check 10 percent of the project data by performing a total review from raw to final results. This activity will occur in addition to the routine management review of all data. Records will be kept to show which data have been reviewed in this manner.

10.0 CALCULATION OF DATA QUALITY INDICATORS

10.1 Precision

Duplicates will be performed on separate, as well as on the same sample source, depending on the method employed. In addition, the final result for a given test may be the arithmetic mean of several determinations on the part or matrix. In this case, duplicate precision calculations will be performed on the means. The following calculations will be used to assess the precision between duplicate measurements.

Relative Percent Difference (RPD) = [(C1 - C2) x 100%] / [(C1 + C2) / 2]

where: C1 = larger of the two observations
C2 = smaller of the two observations

Relative Standard Deviation (RSD) = (s/y) x 100%

where: s = standard deviation
y = mean of replicates.

10.2 Accuracy

Accuracy will be determined as percent recovery of a check standard, check sample or matrix spike.

For matrix spikes and synthetic check samples:

Percent Recovery (%R) = 100% x [(S - U)/T]

where: S = observed concentration in spiked sample
U = observed concentration in unspiked sample
T = true value of spike added to sample.

For standard reference materials (srm) used as calibration checks:

$\% R = 100\% \times (C_m / C_{srm})$

where: C_m = observed concentration of reference material
C_{srm} = theoretical value of srm.

10.3 Completeness

Percent Completeness (%C) = 100% x (V/T)

where: V = number of determinations judged valid
T = total number of determinations for a given method type.

10.4 Project Specific Indicators

Process control limit: range specified by supplier for a given process parameter.

11.0 CORRECTIVE ACTION

11.1 Routine Corrective Action

Routine corrective action will be undertaken in the event that a parameter in Table 9 or Table 10 is outside prescribed limits specified in these tables, or when a process parameter is beyond specified control limits. Examples of non-conformances include invalid calibration data, inadvertent failure to perform method specific QA test, process control data outside specified control limits, failed precision and/or accuracy indicators, and so on. Such non-conformances will be documented on a standard laboratory form. Corrective action will involve taking all necessary steps to restore a measuring system to proper working order and summarizing the corrective action and results of subsequent system verifications on a standard form. Some non-conformances will be detected while analysis or sample processing is in progress, and can be rectified in real time at the bench level. Others may be detected only after a processing trial and/or sample analysis are completed. Typically these types of non-conformances are detected at the Laboratory Manager level of data review. In all cases of non-conformance, sample re-analysis will be considered as one source of corrective action by the Laboratory Manager. If sufficient sample is not available, or the holding time has been exceeded, complete re-processing may be ordered to generate new samples if a determination is made by the technical project leader that the non-conformance jeopardizes the integrity of the conclusions to be drawn from the data. In all cases, a non-conformance will be rectified before sample processing and analysis continues. If corrective action does not restore the production or analytical system causing a deviation from the Project QA Plan, *CTC* will contact the EPA Project Contract Officer. In cases of routine non-conformance, EPA will be notified in the Program Manager's or technical project leader's regular report to the EPA Project Contract Officer. A complete discussion will accompany each non-conformance.

11.2 Non-Routine Corrective Action

While not anticipated, activities such as internal audits by the facility QA engineer, and on-site visits by the EPA Project Contract Officer, may result in findings which contradict deliverables in the Project QA Plan. In the event that non-conformances are detected by bodies outside the laboratory organizational unit, as for routine non-conformances, these problems will be rectified and documented prior to processing or analyzing further samples or specimens.

12.0 QUALITY CONTROL REPORTS TO MANAGEMENT

As shown on the project organization chart in Figure 5, *CTC* maintains a staff of full-time QA engineers who are independent from the project management team. It is the responsibility of the QA engineer to monitor *CTC* Demonstration Projects for adherence to project specific QA Plans. The Laboratory Manager monitors the operation of the laboratory on a daily basis and provides comments to the QA engineer to facilitate his activities. The QA engineer will audit the operation records, laboratory records and laboratory data reports during testing and provide a written report of his findings to the project technical leader and to the Laboratory Manager. The project technical leader will ensure that these reports are included in his report to EPA. The Laboratory Manager will be responsible for achieving closure on items addressed in the report. Specific items to be addressed and discussed in the QA report include the following:

- General assessment of data quality in terms of specific QA objectives in Section 4.1
- Specific assessment of data quality in terms of quantitative and qualitative indicators listed in Section 4.2 and 4.3
- Listing and summary of all non-conformances and/or deviations from QA Plan
- Impact of non-conformances on data quality
- Listing and summary of corrective actions
- Results of internal QA audits
- Closure of open items from last report or communications with EPA in current month
- Deviations or changes in the Project QA Plan
- Progress of *CTC* QA Programs in relation to current project
- Limitations on conclusions, use of the data
- Planned QA activities, open items for next reporting period.

APPENDIX A

Standard Test Product

APPENDIX B

Measurement Locations

APPENDIX C

Apparatus Set-Up

APPENDIX D

Equipment Testing Location

APPENDIX E

ASTM Methods

ASTM B 117 -- Standard Test Method of Salt Spray (Fog) Testing

ASTM B 499 -- Standard Test Method for Measurement of Coating Thickness' by the Magnetic Method: Non-magnetic Coatings on Magnetic Basis Metals

ASTM B 767 -- Standard Guide for Determining Mass Per Unit Area of Electrodeposited and Related Coatings by Gravimetric and Other Chemical Analysis Procedures

ASTM D 522 -- Standard Test Methods for Mandrel Bend Test of Attached Organic Coatings

ASTM D 523 -- Standard Test Method for Specular Gloss

ASTM D 1729 -- Standard Practice for Visual Evaluation of Color Differences of Opaque Materials

ASTM D 1735 -- Standard Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus

ASTM D 2244 -- Standard Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates

ASTM D 2794 -- Standard Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)

ASTM D 3359 -- Standard Test Methods for Measuring Adhesion by Tape Test

ASTM D 3363 -- Standard Test Method for Film Hardness by Pencil Test

ASTM D 3451 -- Standard Practices for Testing Polymeric Powders and Powder Coatings

ASTM D 4060 -- Standard Test Methods for Abrasion Resistance of Organic Coatings by the Taber Abraser

ASTM D 4752 -- Standard Method for Measuring MEK Resistance of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub

ASTM D 5965 -- Standard Test Methods for Specific Gravity of Coating Powders

ASTM G 26 -- Practice for Operating Light Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials